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TECHNICAL
REPORT

WHITE OAK LABORATORY

DEVELOPMENT OF AN IMPROVED SEPARATOR MATERIAL FOR ALKALINE SILVER-ZINC BATTERIES

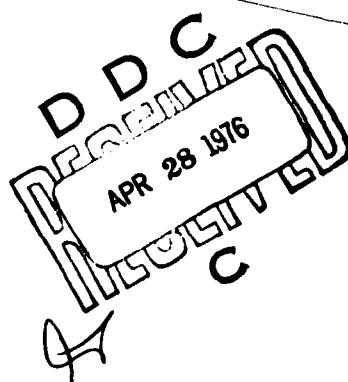
BY

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1 FEBRUARY 1977

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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This work was a preliminary investigation to determine if polyphenylquinoxaline (PPQ) or a codispersed mixture of polyvinylpyrrolidone (PVP) with PPQ could be used to replace cellophane as a separator in the zinc-silver oxide battery. The primary purpose was to compare its dimensional and chemical stability with cellophane and to determine if methods could be developed to lower its high inherent electrical resistance. Dimensional measurements | | |

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were made in 45% KOH at 298 K (25°C) and 353 K (80°C).
Chemical stability to oxidation was observed in KOH solution
saturated with silver oxide at 353 K.

In an effort to lower the resistance of these thin film
membranes, various methods were studied to develop porosity.
Included among these were extraction of the PVP in boiling water,
pyrolysis of the PVP, swelling of the films with phenol, and
precipitation of the PPQ from acetone. Increasing the molecular
weight of the PVP appears very promising and additional studies
are recommended since these polymers have good potential use
as separators.

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1 February 1977

Development of an Improved Separator Material for
Alkaline Silver-Zinc Batteries

This investigation was undertaken to develop a more chemically stable separator for the silver oxide-zinc secondary battery. The separator consists of a codispersed mixture of polyphenylquinoxaline (PPQ) and polyvinylpyrrolidone (PVP). The chemical stability of this separator has been shown to be far superior to commercial cellulose separators. This work is being performed under NAVSEA Task Number SF43431302.

J. R. DIXON
By direction

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INTRODUCTION

Batteries based on the AgO-Zn electrochemical couple have many attractive features for Navy applications. Even though the batteries have not yet been optimized (several aspects require more R & D effort) they have already found acceptance as power sources in missiles, torpedoes, torpedo targets and undersea vehicles. Silver oxide-zinc batteries are built in both primary and secondary configurations. They have high rate discharge capability at a high, fairly constant voltage level and offer the highest energy density of any high rate system (primary or secondary) in use today.¹

One of the essential components of a AgO-Zn battery is the separator. Requirements for a good separator are: chemical resistance to strong alkali and to oxidation by silver oxides, selective porosity, good wetting characteristics, and sufficient strength, flexibility and dimensional stability to withstand the dynamic conditions in the battery environment during cycling. Its primary functions are to allow passage of conducting ions but yet prevent direct mixing of the anolyte and catholyte.²

One of the problems associated with the separators currently used is the chemical stability of the material. Cellophane, (a regenerated form of cellulose) often used in AgO-Zn batteries, is chemically unstable in the AgO-Zn battery environment. The battery electrolyte consists of 45% KOH and contains dissolved oxides of silver. Typical battery operating temperatures for military applications is 353 K (80°C). At these high temperatures, cellophane undergoes oxidative degradation in one of two ways: cleavage of glycosidic linkages which results in depolymerization or by reaction with hydroxyl groups resulting in the formation of aldehyde and carboxyl groups.² Other separator materials, such as Visking sausage casing and "Cellgard" have somewhat better but not entirely satisfactory stability.

1. R. Jasinsky, "High Energy Batteries", Plenum Press, 1967.
2. C. Evans, "Characterization of Cellophane as an Ionic Barrier," NOLTR 63-225, Naval Surface Weapons Center, White Oak, Maryland, March 1964.

Polyphenylene oxide,³ polyethylene,⁴ and inorganic separators⁵ are presently being evaluated as replacement separators in the AgO-Zn battery. At this center, polyphenylquinoxalines (PPQ), which have been under development since 1968, are known to have excellent chemical stability in hot alkali.

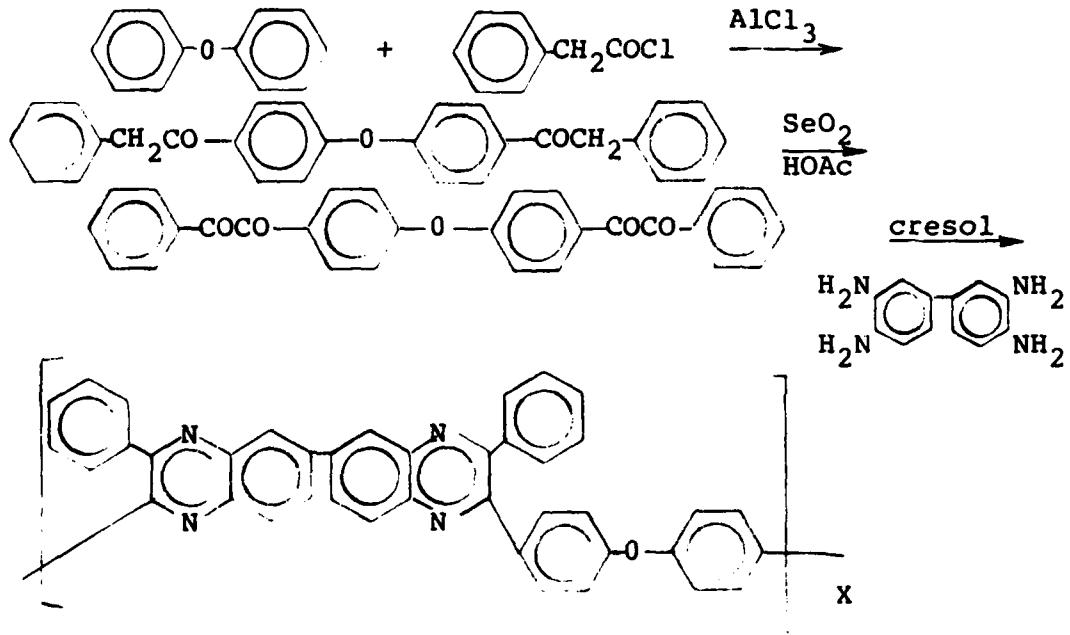
The primary purpose of this investigation was to determine if a method was available to decrease the inherent electrical resistance of the PPQ without affecting its chemical stability.

EXPERIMENTAL

Polymer Preparation

Poly-2,2'-(p,p'oxydiphenylene)-6,6'-di-(3-phenylquinoxaline)

The PPQ polymer used in the preparation of the membranes used in this study was purchased as a 10% solution in m-cresol from the Narmco Division of the Whittaker Corporation. The synthetic route used to prepare this polymer is as follows:



3. R. Walker and S. Zaman, 149th ECS Meeting, Washington, D.C., May 1976.
4. V. D'Agnostino, J. Lee and G. Orban, "Zinc-Silver Oxide Batteries," Ed., A. Fleischer and J. Lander, Chapter 19, J. Wiley, 1971.
5. G. Moe and F. Arrance, "Zinc-Silver Oxide Batteries," Editors, A. Fleischer and J. Lander, Chapter 21, J. Wiley, 1971.

The reported inherent viscosity for this polymer is 2.05 (dl/g) and the glass transition temperature is 693 K (420°C).⁶

Membrane Preparation

Membranes for resistance measurements were prepared by spreading the polymer solution onto a glass plate (16 cm x 28 cm x 9 mm) with a metal bar. The thickness of the final film was controlled by means of masking tape which was placed along the edges of the glass plate. It was found necessary to thoroughly wash the glass plate with water and detergent and then to rinse with isopropyl alcohol to insure good wetting by the polymer solutions.

Following the casting of the film, the solvent (m-cresol) was removed under vacuum at elevated temperature. Care must be taken to insure that the glass plate remains level during this operation if uniform thickness is to be obtained. The glass plate was allowed to equilibrate at 333 K (60°C) under vacuum before final drying at 398 K (125°C).

After cooling, the film was removed from the plate by means of a razor blade. In difficult cases it may be necessary to use enough water to assist in "lifting" the film from the plate.

The codispersed polymer solutions were prepared by first dissolving polyvinylpyrrolidone (PVP)* in m-cresol at 353 K (80°C) and then adding this solution with stirring to a heated PPQ solution 353-363 K (80-90°C). Codispersed membranes were prepared in the same manner described for PPQ films.

Stability Measurements

Dimensional

Rectangular pieces (approximately 50 x 60 cm) of membranes were cut and accurately weighed after equilibration to ambient laboratory conditions. The sample was then accurately measured with calipers and a micrometer to determine its true dimensions.

The specimens were then placed in vials which contained 45% by weight of KOH and exposed either at ambient or 353 K (80°C). The specimens were removed from the alkali solution at regular intervals and, after "blotting" the excess solution, were again accurately measured and weighed. The results were recorded over a period of 28 days and are reported as percent change.

Chemical

The chemical stability in the silver-zinc battery environment was evaluated in a qualitative fashion by visual observation of

6. P. Hergenrother and H. Levine, *J. Polymer Science*, A-1, 5, 1453-1466 (1967).

*GAF Corporation

exposed specimens. Samples (0.5 x 3 cm) were cut and immersed in a 45% solution of KOH which had been saturated with AgO. The samples were placed in an oven at 353 K (80°C) and removed at regular intervals for inspection. Samples which had changed color or had become deformed or brittle were judged to be chemically unstable.

Thermal

The thermal stability of PPQ and PVP were determined in vacuum using an Ainsworth recording balance. The sample (~10 mg) was weighed into a crucible and placed in a quartz tube which is heated by means of a Marshall furnace. The temperature of the apparatus is raised at a rate of 279 K (6°C)/minute and the vacuum is approximately 0.1 mm. The sample weight is continuously recorded with increasing temperature up to 1073 K (800°C). The results are plotted as temperature versus percent weight loss.

Resistance Measurements

Fisher Scientific 45% KOH was used as received in all the studies. A lucite diffusion cell, similar in design to that used by Lander,⁷ was used to measure the high membrane resistance in the initial development stages. Leakage around the Teflon membrane holder was found nonexistent. This was checked by placing nonporous plastic sheets varying from .0012 to .010 cm (.5 to 4 mil) thickness between the teflon and measuring the AC electrical resistance of a 45% KOH solution. The resistance was greater than 10 megohms. A second test involved placing water containing phenolphthalein on one side, with KOH on the other side of the plastic insert. No visual coloration appeared in the KOH on standing 24 hours.

For lower resistance membranes, a cell obtained from RAI Research Corporation was used. This cell contained 1 square inch platinized platinum electrodes approximately .25 cm apart, with a cell constant of $3.87 \times 10^{-2} \text{ cm}^{-1}$.

Resistance measurements were made using a General Radio 1650 B impedance bridge with a 219 K General Radio external capacitor decade box connected to the bridge by shielded cables and adjusted in steps of 10^{-4} microfarads to give the best resistive balance.

The thickness of the membranes was measured by a micrometer in several locations of the film and the average taken.

DISCUSSION OF RESULTS

Polyphenylquinoxaline (PPQ) is a nonpolar polymer, hydrophobic to the KOH electrolyte, with a high inherent electrical resistance. In order to reduce the electrical resistance, polyvinylpyrrolidone (PVP),

7. J. Lander, Characteristics of Separators for Alkaline Silver Oxide-Zinc Batteries, J. Cooper and A. Fleischer, Characteristics of Separators for Alkaline Silver Oxide-Zinc Batteries, Chapter 11.

which is polar and hydrophilic, was incorporated to form a codispersed heterogeneous membrane. The idea was to remove the polar PVP polymer by extraction of this soluble phase in hot water or by vacuum pyrolysis of the PVP leaving behind a semi-porous film consisting of the PPQ matrix with an unknown amount of PVP remaining.

We began by adding 1 and 5% PVP to the PPQ. Dimensional stability studies of these codispersed films and pure PPQ were compared with Pudo cellophane. The length and width measurements were not distinguished, and after soaking in 45% KOH, the percentage dimensional changes as a function of time at 298 K (25°C) and 353 K (80°C) are shown in Figures 1 and 2 respectively. Similar dimensional changes in the films thickness versus exposure time in 45% KOH at 298 K (25°C) and 353 K (80°C) is shown in Figure 3. Much of the scatter is due to measuring different sites on a film of non-uniform thickness. As expected, the hydrophilic cellophane has absorbed KOH electrolyte to more than double its thickness. This is also made apparent in Figure 4 where the percent weight change of films soaked in KOH at 298 K (25°C) and 353 K (80°C) is plotted as a function of exposure time. The large percentage weight increase in cellophane compared to PPQ or PVP-PPQ films is in agreement with its hydrophilic nature and the dimensional change in its thickness.

Calculations of separator porosity from resistance measurements indicated relatively little porosity (1-2%) in these PVP-PPQ films. We increased the % PVP in the PPQ-PVP film and found no changes in the dimensional or chemical stability of the films. Chemical stability studies were performed by placing PVP-PPQ films along with cellophane in 45% KOH saturated with AgO at 353 K (80°C). After one day the cellophane was badly oxidized whereas the PVP-PPQ films showed no oxidative damage after one week.

Since the PPQ polymer does not absorb KOH to swell sufficiently to allow passage of conducting ions, a method to create porosity in the PVP-PPQ film had to be devised. A summary of the methods is included in Table I.

Removal of the PVP from the PVP-PPQ composite film by extraction in boiling water to create voids in the film was determined by resistance measurements. These initial high resistance measurements were carried out using the lucite diffusion cell. The ability of this cell to function in accordance with the equation

$$R = K \frac{1}{A} \quad (1)$$

was determined by measuring the resistance of membranes of various thickness. The necessary linearity of resistance, R, with thickness, l, is illustrated by the graph in Figure 5.

The original pure PPQ polymer film has a high resistance of approximately 10 Kohm. Addition of various percentages of low molecular weight PVP followed by extraction in boiling water appeared to be effective in creating some porosity. This is illustrated by

the graph in Figure 6 where increasing percentage PVP is shown to decrease the resistance of the separator film.

Before thermal analysis data was available to determine the decomposition temperatures of the PVP, PPQ and PVP-PPQ films, a 0.0038 cm (1.5 mil) thick 43% PVP-57% PPQ membrane was heated at 598 K (325°C) in air for 2 hours and the resistance was determined to be 154 ohms. Similarly, heating a 60% PVP-40% PPQ 0.0012 cm (0.5 mil) film in N₂ at 598 K (325°C) gave a reduced resistance of 440 ohms. At this stage of the work, heating a PVP-PPQ film in air appeared to reduce the resistance almost two orders of magnitude from the pure PPQ film. No visible oxidative damage appeared. Both of these films were made from the 40,000 molecular weight PVP.

The third technique for creating porosity in the film was used in conjunction with the water extraction. After some of the PVP was leached out in the hot water, the remaining composite PVP-PPQ matrix was allowed to soak in a saturated solution of phenol in water at 298 K (25°C). The phenol is a plasticizer creating internal free volume in the PVP-PPQ matrix. The effects of the phenol swelling on the dimensions of the film is shown in Table II. The large weight increase and thickness increase is indicative of free volume in the film.

Thermal analysis of PVP, PPQ and a composite 40% PVP-60% PPQ films are illustrated by the graphs in Figure 7. Thermal decomposition of pure PVP begins to occur in the vicinity of 648 K (375°C), above our previously selected 598 K (325°C). The decomposition of PPQ begins at 823 K (550°C). The PVP-PPQ film shows decomposition temperatures of 648 K (375°C) and 823 K (550°C) in agreement with decomposition of a composite heterogeneous mixture. Using a low molecular weight (10K) PVP four PVP-PPQ membranes were prepared containing 10, 20, 40 and 60% PVP. Pyrolysis of these films in a vacuum above the PVP decomposition at 698 K (425°C) gave higher resistances than expected. Resistance of 0.0025 cm (mil.) thick membranes were 1980 and 1261 ohms for the 10 and 20% PVP films, respectively. The 40 and 60% PVP films developed visible holes due to removal of PVP. The fact that the membranes heated at 598 K (325°C) gave lower resistances than those heated at 698 K (425°C) can not be immediately explained.

The most plausible explanation for the fact that higher membrane resistances are obtained when the carbonization temperature is 698 K (425°C) versus 598 K (325°C) is that at the upper decomposition temperature the matrix polymer (PPQ-1) is above its glass transition temperature, 693 K (420°C). The voids which form as the PVP decomposes at 425°C would be transient in nature and subject to the viscoelastic "flow" of the PPQ-1 matrix phase. At 598 K (325°C) the voids which are created are permanent since the matrix phase is in its glassy state and not able to undergo viscoelastic "flow".

Comparison of various methods for creating membrane porosity is represented in Table III as a function of molecular weight of the PVP. The resistance measurements represent a median value for several measurements of different films. The resistance decreased with increasing molecular weight of the PVP incorporated into the composite PVP-PPQ film.

At the conclusion of this preliminary study, we measured the resistance of a pure PPQ film precipitated by extraction of the m-cresol solvent with acetone. The results are similarly expressed in Table III. The specific resistance of this film was an order of magnitude lower than that of previous membranes.

CONCLUSIONS

Starting with a very high resistance film of pure PPQ we have shown that a composite PVP-PPQ film has excellent dimensional and chemical stability to hot KOH saturated with AgO. The resistance can be reduced by several methods, namely, extraction of the PVP in water, pyrolysis of the PVP at elevated temperatures and adding a phenol plasticizer. Additionally, the precipitation of PPQ with acetone appears to be another method with good promise for creation of internal volume.

Future Plans

Future work will include studies on the effect of higher molecular weight PVP (180,000 and 300,000) on the resistance of the PPQ-PVP separators. Methods for pyrolyzing higher percentage PVP separators at 698 K (425°C) without developing holes will also be studied. Variation of the PPQ concentration in the m-cresol solvent for the acetone precipitation of PPQ will be investigated to produce thinner porous films.

The final goal is to combine the results of several methods of increasing porosity to produce a separator having low electrical resistance, good chemical stability and ion selectivity.

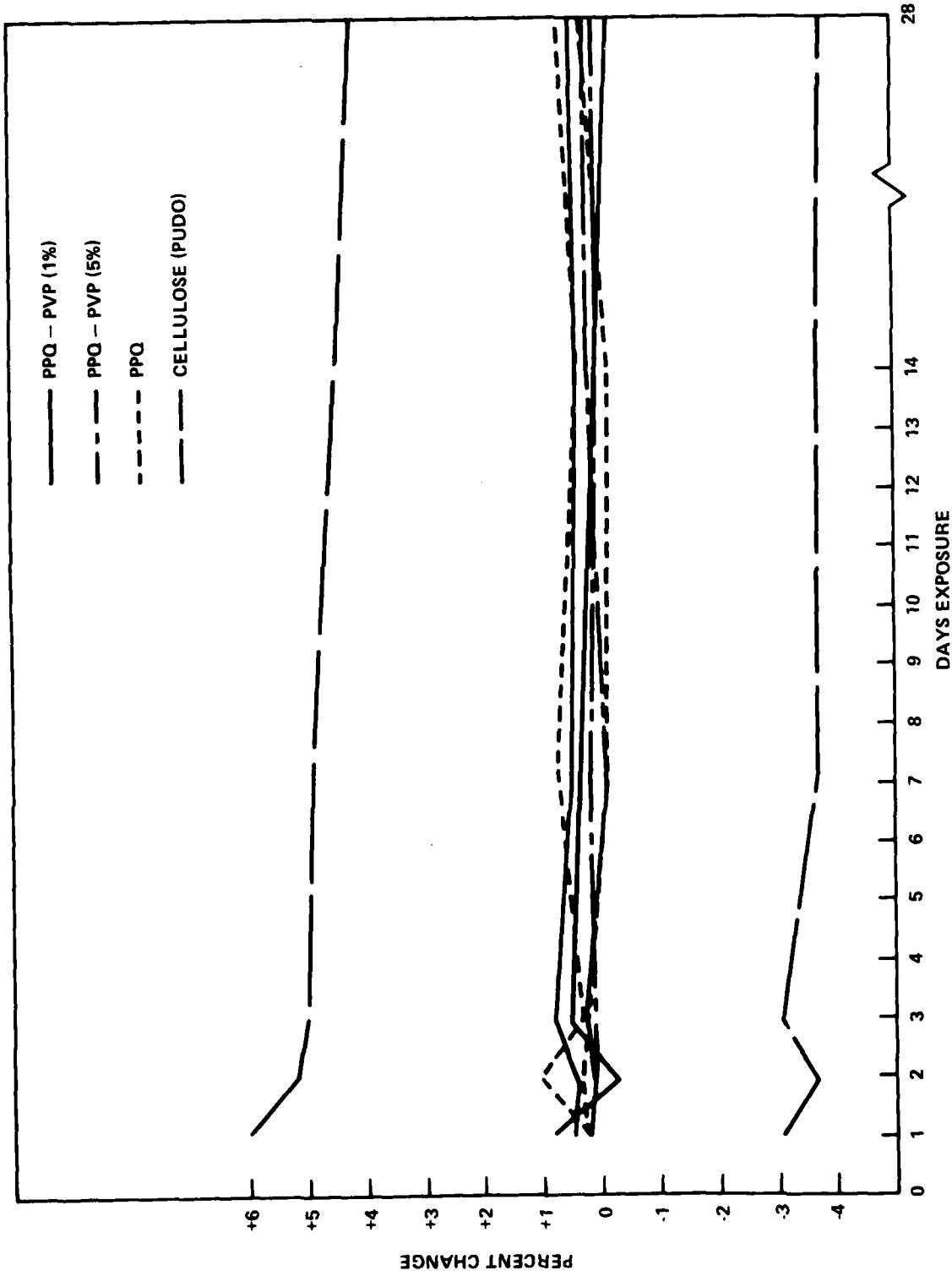


FIG. 1 DIMENSIONAL CHANGE (LxW) VS EXPOSURE TIME IN
45% KOH AT 298 K (25°C)

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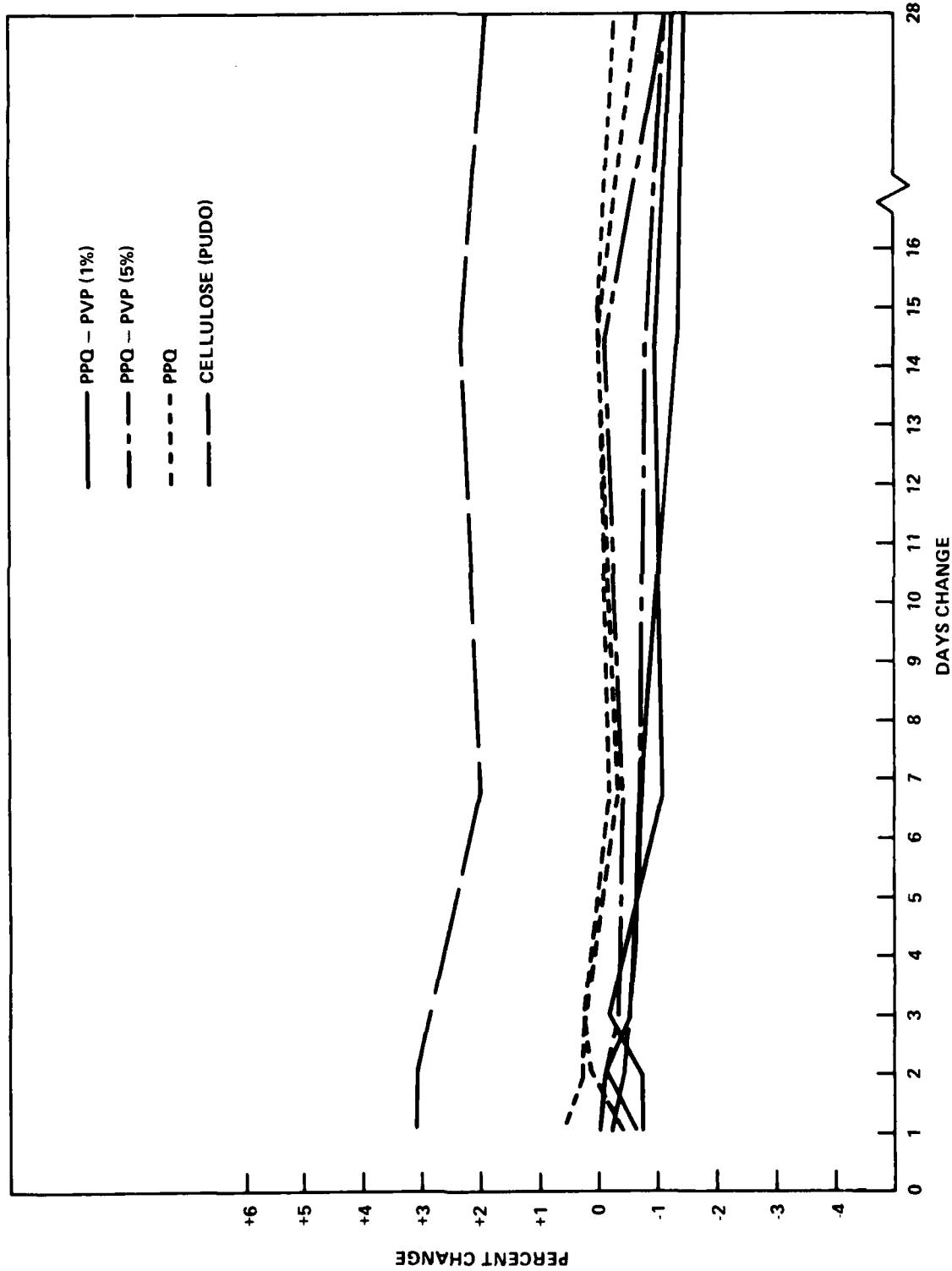


FIG. 2 DIMENSIONAL CHANGE (L_{XW}) VS EXPOSURE TIME IN
45% KOH AT 353 K (80°C)

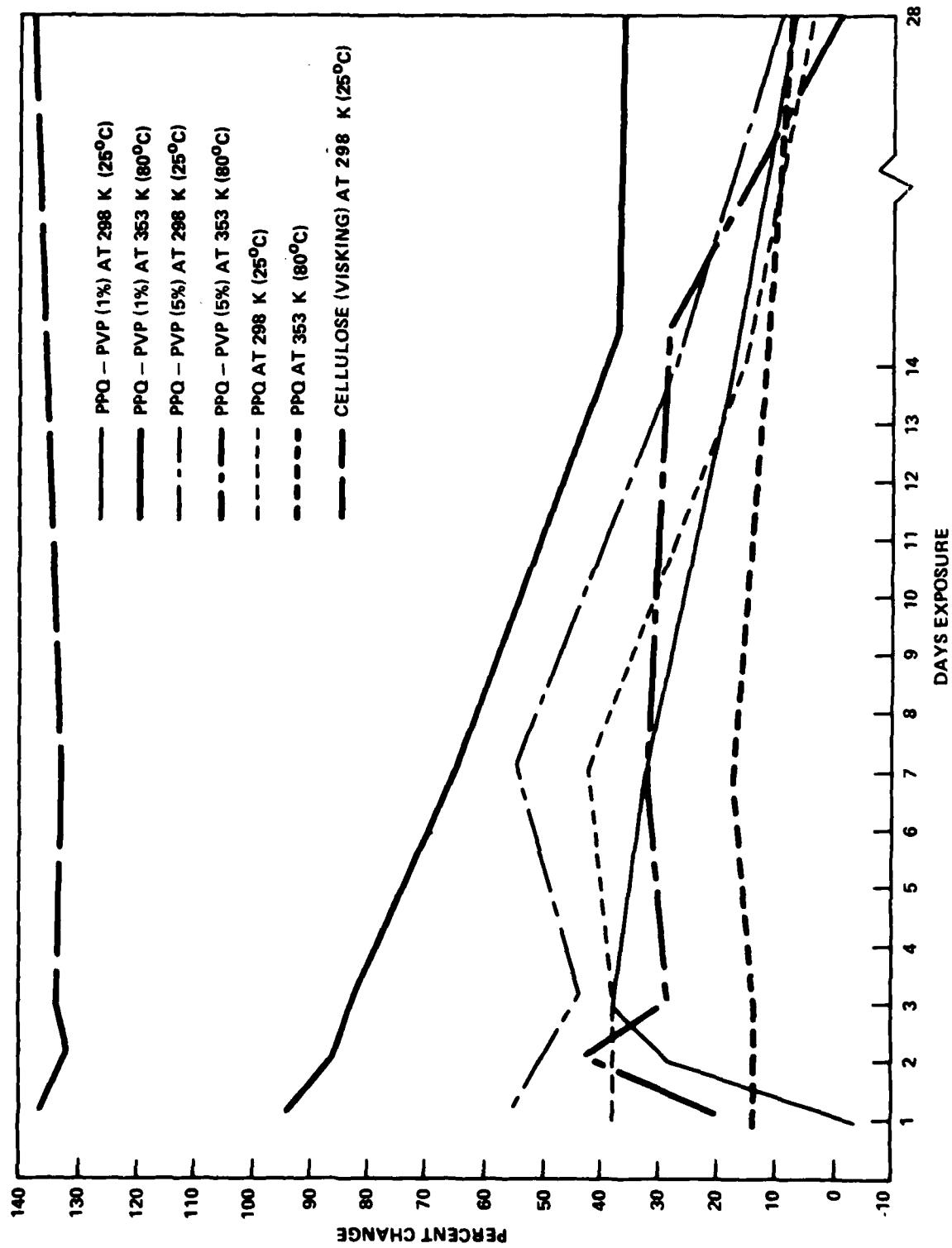


FIG. 3 DIMENSIONAL CHANGE (THICKNESS) VS EXPOSURE TIME
IN 45% KOH AT 298 K (25°C) AND 353 K (80°C)

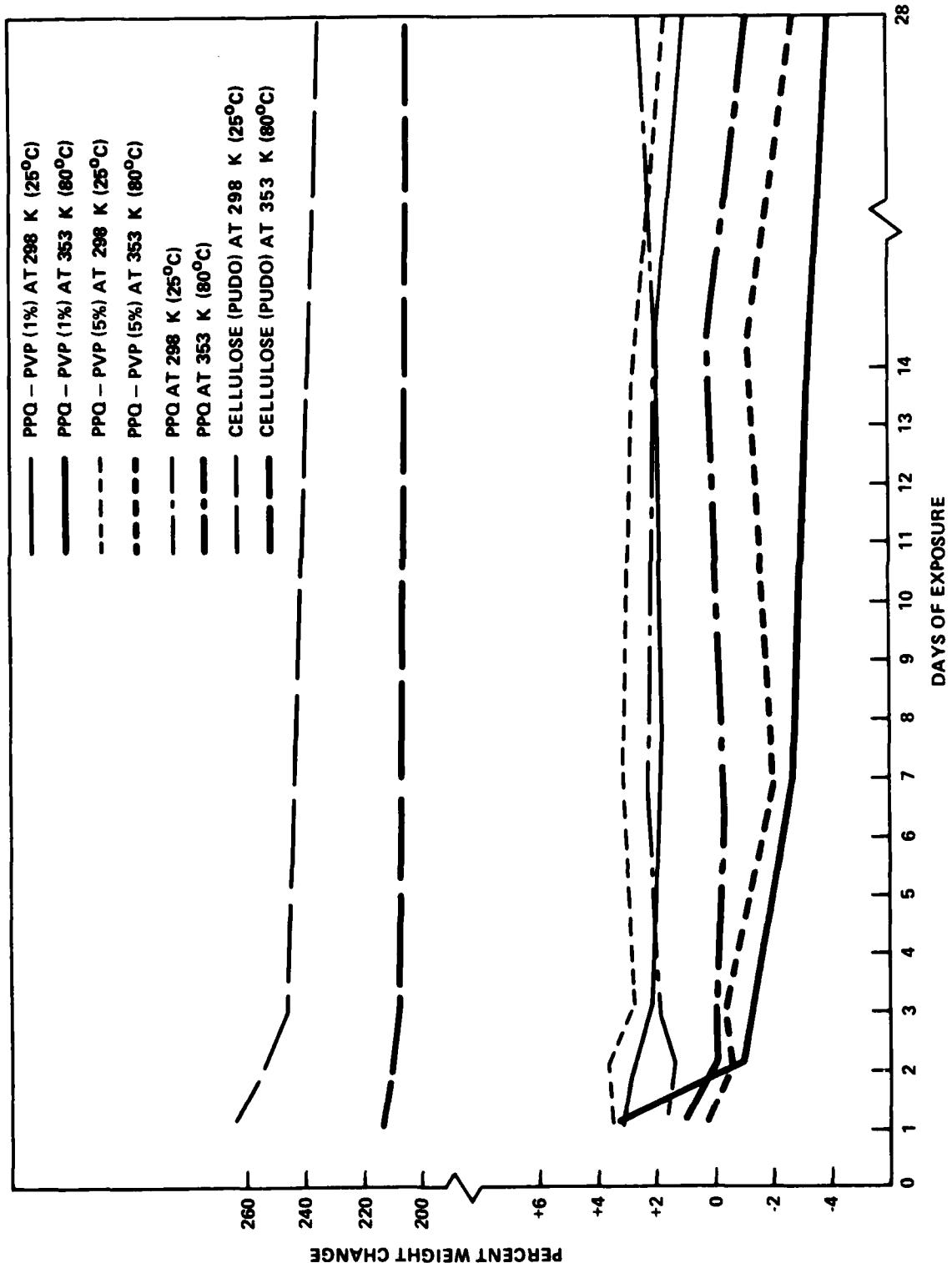


FIG. 4 WEIGHT CHANGE VS EXPOSURE TIME IN 45% KOH AT 298 K (25°C) AND 353 K (80°C)

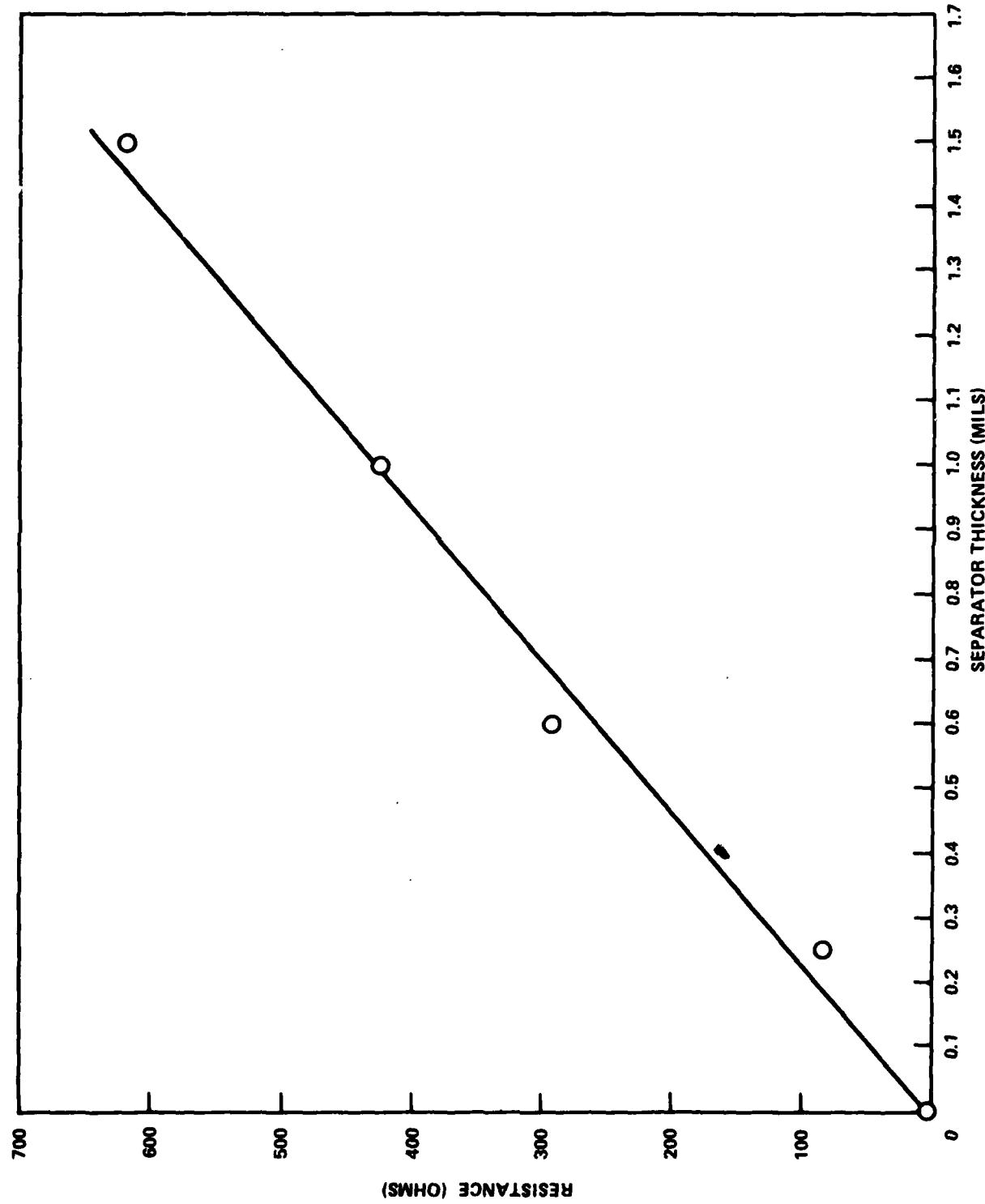


FIG. 5 RESISTANCE OF A 40% PVP - 60% PPO SEPARATOR IN 45% KOH AT 298 K (25°C) VS SEPARATOR THICKNESS

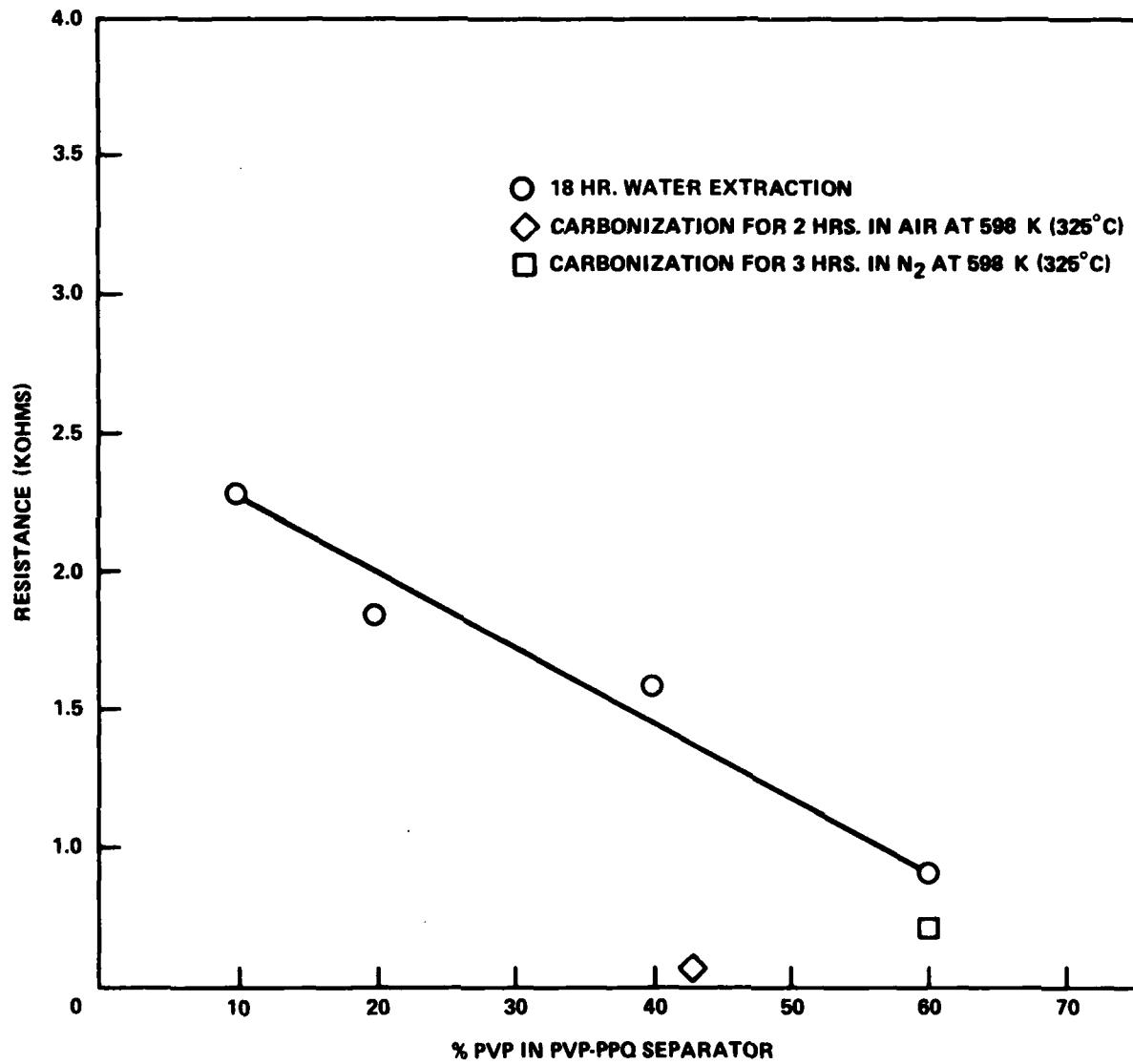


FIG. 6 RESISTANCE VS % PVP IN PVP-PPQ SEPARATORS IN 45% KOH
AT 298 K (25°C)

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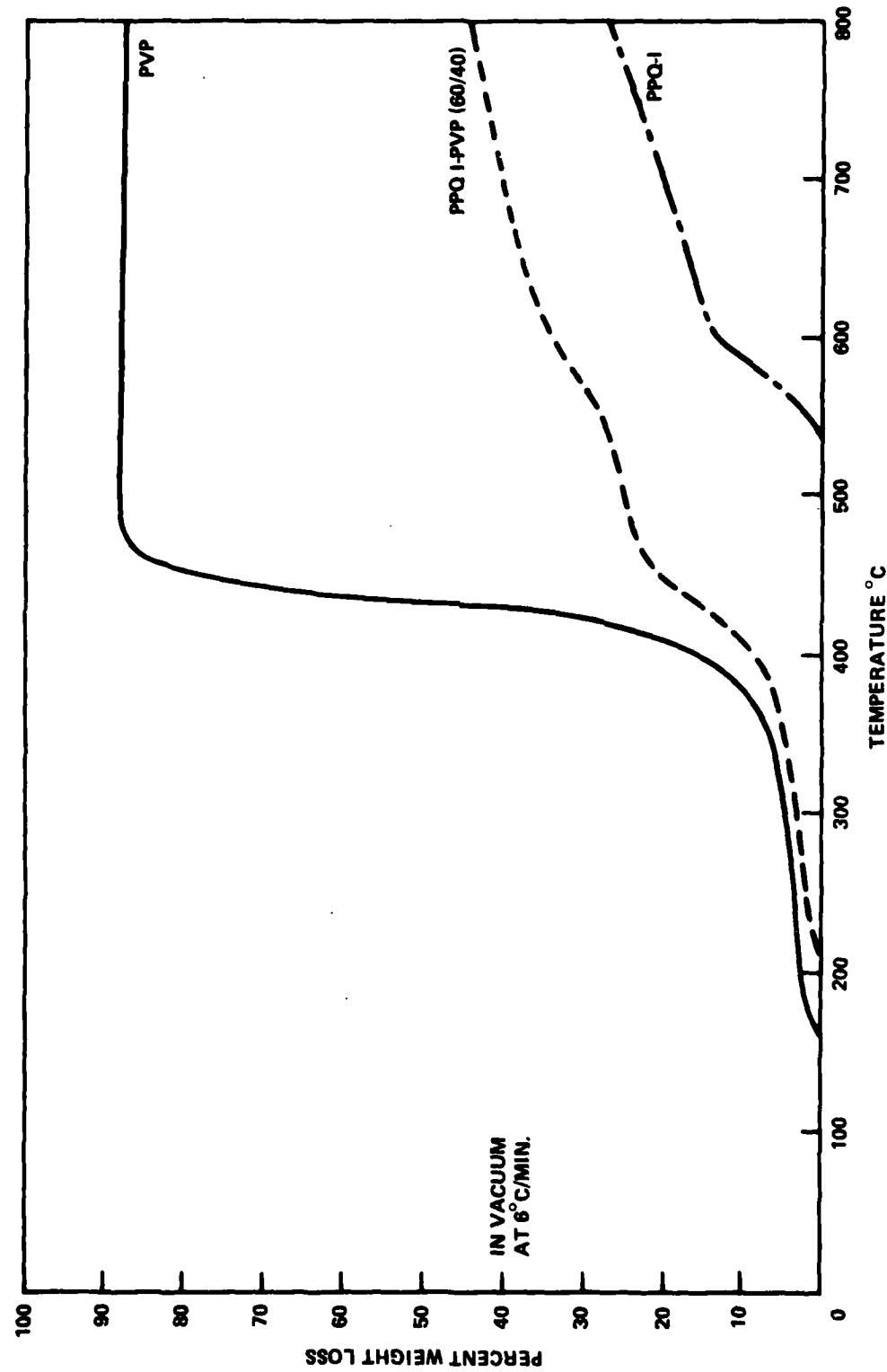


FIG. 7 THERMOGRAVIMETRIC ANALYSIS OF PVP (K15) AND PPO

TABLE I METHODS FOR DEVELOPING
POROSITY IN PPQ MEMBRANES

- A. CODISPERSED PVP-PPQ FILMS
 - 1. EXTRACTION OF PVP IN WATER
 - 2. CARBONIZATION OF PVP
 - 3. SWELLING PPQ WITH PHENOL
- B. PRECIPITATION OF PPQ
 - 1. ACETONE EXTRACTION OF CRESOL

TABLE II SWELLING OF PVP-PPQ IN PHENOL-WATER

FILM COMPOSITION: 57% PPQ - 43% PVP

CONDITIONS: 8% AQUEOUS PHENOL AT 298 K (25°C)

| SOAK TIME (HRS.) | WEIGHT (GMS.) | THICKNESS (MILS.) | LENGTH (MM.) | WIDTH (MM.) |
|---------------------|-------------------|----------------------|------------------|------------------|
| 0 | 0.06571 | 1.2 - 1.4 | 57.44 | 36.44 |
| 16 | 0.12600 (+92%) | 1.8 - 2.3 | 58.85 (+2.4%) | 37.10 (+1.8%) |

TABLE III COMPARISON OF RESISTANCES AS A FUNCTION OF PVP
MOLECULAR WEIGHT AND PORE FORMATION TECHNIQUE

| <u>PVP MOL WT. = 10,000</u> | | | |
|-----------------------------|--|--------------------------------|------------------------------|
| % PVP | <u>MEMBRANE THICKNESS (MILS)</u> | <u>MEMBRANE TREATMENT</u> | <u>RESISTANCE (OHMS)</u> |
| 40 - 45 | 1.0 | H ₂ O EXTRACTION | 1590 |
| <u>PVP MOL WT. = 15,000</u> | | | |
| 40 - 45 | 1.0 | H ₂ O EXTRACTION | 289 |
| 40 - 45 | 1.0 | H ₂ O - PHENOL SOAK | 9 |
| <u>PVP MOL WT. = 40,000</u> | | | |
| 40 - 45 | 1.0 | H ₂ O EXTRACTION | 46 |
| 40 - 45 | 1.0 | H ₂ O PHENOL SOAK | 6 |
| 40 - 45 | 1.5 | CARBONIZATION 598 K (AIR) | 150 |
| IV | | | |
| 0 | 13 | ACETONE PRECIPITATED PPO | 4 |

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